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Synthetic Organic Chemicals

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The Preparation of Synthetic Organic Chemicals at Kodak Park

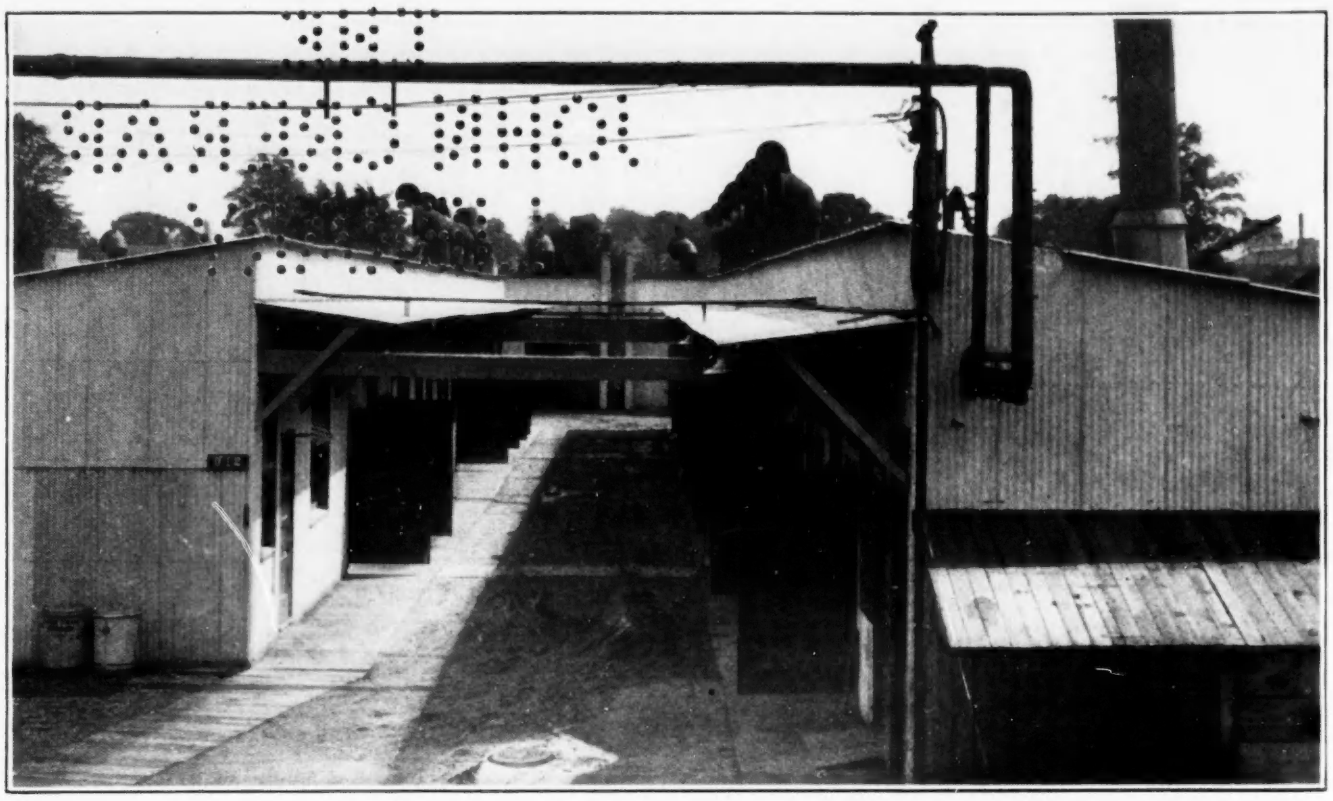
IN order to carry on research in organic chemistry, a chemist needs a supply of the raw materials from which the compounds on which he is working are to be built up. As a general rule, the synthesis of the compounds in which he is interested will involve a considerable number of steps before they can be produced from those which are available from the industries directly. The first step is the purification of the technical materials produced by the industries, and while the investigator can, of course, perform all these operations for himself such a procedure involves considerable loss of time, and it is very advantageous for him to be able to obtain materials as near as possible to those which he requires for the final synthesis. The supply of synthetic organic chemicals, therefore, involves the collection from the industries of all the materials which are available, in most cases the purification of those materials so as to render them fit for further synthetic work, and, finally, the synthesis of a considerable number of intermediates made from them in order to carry the work at least one step, if not more, nearer to the stage where the research worker can utilize the material for his work.

In 1918 the Eastman Kodak Company decided to undertake the production of synthetic organic chemicals with a view to manufacturing the materials required for research in organic chemistry, and to making as complete a line of those materials as possible available to American chemists.

The decision of the company was received with enthusiasm by the American Chemical Society and by all the more important organic chemical manufacturing firms, who placed at the disposal of the undertaking not only their finished products but the intermediates which they prepare exclusively for their own use.

The work undertaken may be divided into three sections. Of primary importance is the synthesis of compounds which are not prepared technically but are required for laboratory purposes; secondly, the purification of technical materials obtained from the chemical manufacturers; and, thirdly, the distribution of such technical chemicals in the form in which they are purchased.

By undertaking this last duty the laboratory was of service not only to chemists and the purchasing agents of universities, but also to the manufacturers themselves, since to them retail sales in small quantities present great difficulties, and yet, in the public interest they did not care to refuse to supply such small quantities. An understanding was reached between the manufacturers and the laboratory of the Eastman Kodak Company whereby inquiries for small



A View of the Synthetic Organic Laboratories at Kodak Park

quantities of chemicals are referred by the former to the company and inquiries for large amounts are referred by the latter to the manufacturers.

At the present time about 2,300 chemicals are in stock and are listed in the new price list No. 17 which has just been issued.

Many firms supply us not only with products which they manufacture for their own exclusive use, but also in several instances with materials specially prepared for their work or obtained by their research laboratories. Assistance has also been rendered by firms making specialties of a few of the less common products, as well as by certain individual workers in university and other laboratories.

As new products become available, lists of them are published in the advertising section of the News Edition of "Industrial and Engineering Chemistry."

The chemicals are classified in three grades, the Eastman chemicals, the Practical chemicals, and the Technical products. The Eastman chemicals are of the highest purity which it is feasible to obtain. The Practical chemicals, while not pure, are sufficiently so for synthetic purposes. This class also includes a few substances synthesized in the laboratory itself, and in which small amounts of impurities are known to be present, as well as a series of inorganic compounds commonly employed in organic syntheses. The Technical products are the chemicals as obtained from large scale manufacturers who cannot conveniently retail small quantities for experimental work.

The preparation of synthetic organic chemicals at Kodak Park has been carried on successfully for nine years. Processes have been worked out, refined and simplified so that today many of these chemicals cost less than they did in the beginning. This has been quickly appreciated by chemists so that these laboratories are now established as the source from which American research workers draw the supplies which they require.

In order to keep chemists advised of the advances which are being made and also to interest them in various aspects of the subject, it has been decided to publish this leaflet at regular intervals, approximately bi-monthly.

We shall be glad to receive criticisms and suggestions for its improvement.

Some Carbohydrates

EVER since it was shown that bacteria can be classified by their ability or refusal to digest specific sugars, these have occupied an important position in the armory of the bacteriologist. An outline of the manner in which some of the rare sugars are prepared and purified may therefore be of interest.

The most important from a bacteriological point of view is lactose (milk sugar). The commercial material is purified by dissolving in water, treating the solution with purified decolorizing carbon and pouring into a large volume of alcohol.

Next in importance come sucrose, dextrose and xylose, together with the polysaccharide inulin, and the sugar alcohol mannitol. Sucrose, or cane sugar, comes on the market in so high a state of purity that further treatment is unnecessary.

Dextrose, more commonly called glucose, is of especial interest in that it is the sole product of the acid hydrolysis of starch and cellulose. Until recently it was obtainable commercially only as an impure syrup, but as a result of careful manufacturing research by the Corn Products Refining Company, it can now be obtained in practically unlimited quantities as a crystalline powder of very high purity, at a cost comparing favorably with that of cane sugar. While of almost equal nutritive value, it has only half the sweetening power of the latter, and has a lower solubility in water. The commercial product is recrystallized from alcohol to render it suitable for bacteriological purposes.

The pentose xylose is produced by the hydrolysis of xylan, a carbohydrate associated with cellulose in many vegetable tissues. It may conveniently be obtained by warming corn cobs with dilute sulfuric acid.

Inulin is the starch-like component of the Jerusalem artichoke. It is chemically of interest in that its sole product of hydrolysis is levulose (fructose).

Mannitol occurs naturally in manna and is formed by fermentation of cane sugar under certain conditions, as well as by the reduction of mannose.

Many other sugars are employed by the bacteriologist for making fine differentiations. Such are raffinose, a trisaccharide present in cottonseed meal; arabinose, recently shown by Anderson and Sands to be readily obtainable from mesquite gum; mannose, obtained by hydrolysis of vegetable ivory; rhamnose, from the glucoside quercitrin; fucose, from seaweed.

Glycogen, the form in which reserve carbohydrates are stored in the animal body, closely resembles starch. It is prepared by extraction of liver from freshly killed animals with alkali and precipitation of the extract with ethyl alcohol. The first stages of the preparation must be carried out with the greatest possible speed, owing to the presence in the liver of an enzyme which breaks down glycogen into dextrose.

Closely resembling the carbohydrates are the cycloses, of which the most important are the inositols or hexahydroxycyclohexanes. The most important of these is the inactive variety, which was first isolated from animal tissues. It is most conveniently prepared from its hexa-phosphoric acid, phytin, which occurs in corn. Quebrachitol, a monomethyl ether of inositol, could be obtained in practically unlimited quantity from the serum of rubber latex.

Glucosamine consists of dextrose in which one of the hydroxyl groups is replaced by an amino group. This substance is formed by the acid hydrolysis of chitin, an insoluble substance forming the principal component of the shells of lobsters, crabs, and many insects.

Have you received the New List No. 17 of Eastman Organic Chemicals? If not, a request will bring one to you.

New Chemicals

One section of the Synthetic Chemistry Department is devoted to the preparation of new compounds which have been requested from different sources. We are always glad to consider the preparation of chemicals not listed in our catalog, and to give prompt reply as to the possibility of their manufacture.

The following chemicals for which requests have been received were added to our list within the last two months.

* Acetyl m-Toluidine
* Tertiary-Amyl Chloride
* Tertiary-Amyl Iodide
* Benzophenoneoxime
* Cyanoacetamide
* Cyclohexane
m-Diethylaminophenol
* Dipentene
Dipentene (Tech.)
* Ethyl Xanthate
* Iso-Eugenol Methyl Ether
* Nitrosopiperidine
* Succinyl Chloride
Tetramethyldiaminodiphenylmethane
Thionyl Chloride (Practical)
* Allyl Urea
* Dichloroacetamide
2.6-Dichlorophenol
* Ethylamine (anhydrous)
* Tetramethyl Benzidine

An asterisk before an Eastman Organic Chemical indicates that the compound is either prepared or purified in the Kodak Research Laboratories.

PLAN TO ATTEND
THE SECOND NATIONAL
SYMPOSIUM ON
ORGANIC CHEMISTRY
TO BE HELD AT
COLUMBUS, OHIO
DECEMBER 29-31, 1927

Organic Chemicals as Analytical Reagents

I REAGENTS FOR COPPER

α-BENZOINOXIME (Cupron)

Feigl, Ber. *56*, 2083 (1923)

Copper is precipitated by this reagent from hot ammoniacal solution in the form of green copper-benzoinoxime. The precipitate (which is insoluble in water, dilute ammonia, alcohol, or tartrate solution) is washed, dried at 110° and weighed. Copper-benzoinoxime contains 22.02% copper, and on account of its high molecular weight is a more suitable compound for gravimetric analysis than either the sulphide or oxide.

PHENYLTHIOHYDANTOIC ACID

Willard and Hall, J. A. C. S. *44*, 2253 (1922)

Copper may be separated from metals of the iron and zinc groups in the form of a yellow organic compound by precipitation from a solution slightly acidified with acetic acid, by means of phenylthiohydantoic acid. After filtering and washing while hot, the precipitate is ignited to the oxide and fused with potassium pyrosulphate. After dissolving in water, the copper may be determined by any of the volumetric or electrolytic methods.

s-DIPHENYLCARBAZIDE

Kolthoff, Chem. Weekblad, *21*, 20 (1924)

Neutral solutions of many of the metals give characteristic colors when treated with this reagent. As little as one mg. of copper per liter gives a reddish-violet color which may be intensified by extraction with benzene. Large amounts of other metals obscure the test, but the method is very satisfactory where it is known that the solution does not contain other metals.